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(58) Field of search

**C3K**

**C3V**

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**(54) Melt spinning thermoplastic polymers**

**(57) A method of spinning synthetic fiber by the extrusion of thermoplastic polymers, comprising the step of admixing to these polymers, prior to the extrusion step, a stearate-based additive.**

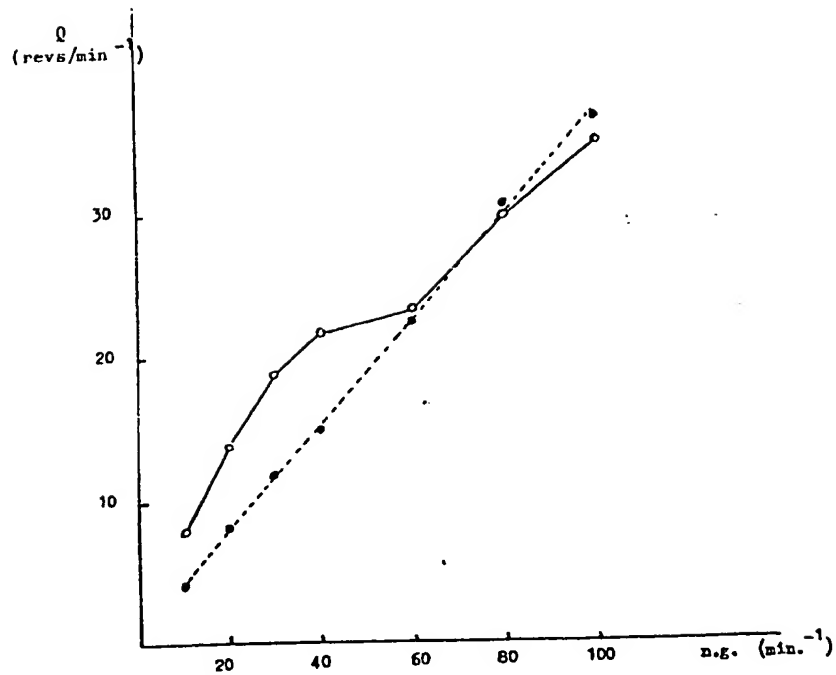
**Fibers obtained by the disclosed method and containing the stearate-based additive in concentrations ranging from 0.03% to 0.3% by weight.**

**GB 2 152 061 A**

1/4

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GRAPH 1

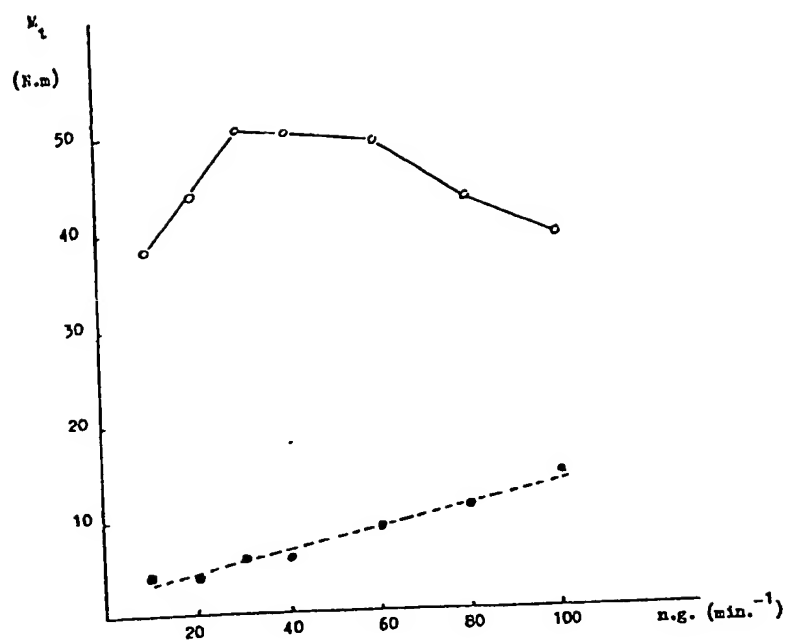


— o Polyethyleneterephthalate as such  
 - - - • Polyethyleneterephthalate with 0.04%  
 stearyl stearate

2/4

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GRAPH 2

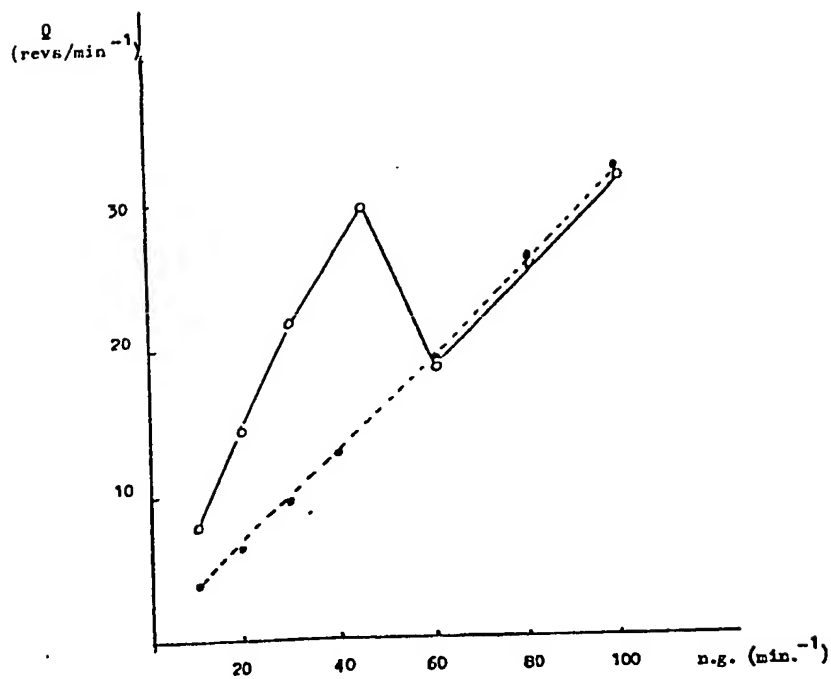


— o Polyethyleneterephthalate as such  
- - - • Polyethyleneterephthalate with 0.04%  
stearyl stearate

3/4

2152061

GRAPH 3

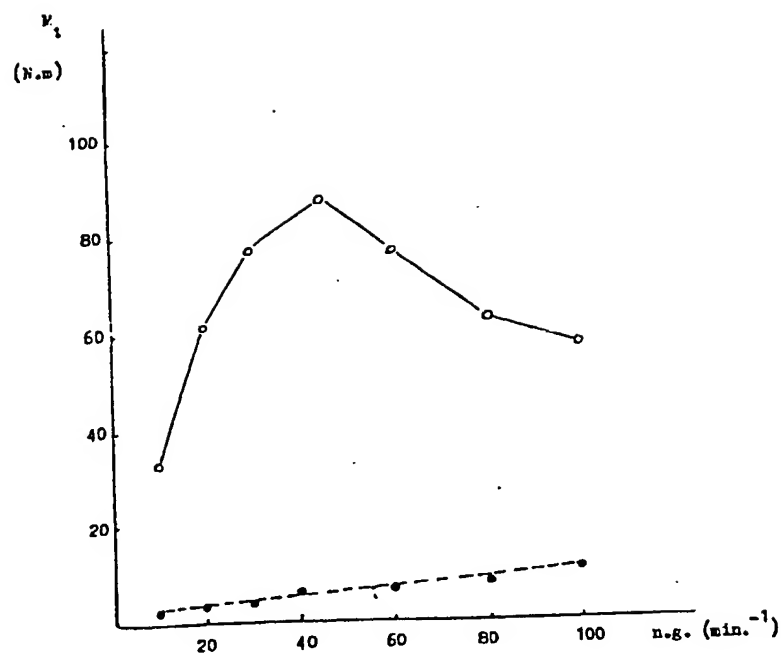


— o Nylon 6 as such  
- - - • Nylon 6 with 0.1% N,N'-ethylenebisdistearamide

4/4

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GRAPH 4



\_\_\_\_\_ o Nylon 6 as such  
 - - - - - • Nylon 6 with 0.1% N,N'-ethylenebisdistearamide

## SPECIFICATION

**A method of spinning thermoplastic polymeric materials, by melting synthetic fibers, and related fiber and articles of manufacture**

- 5 This invention relates to a method of spinning thermoplastic polymeric materials, by melting synthetic fibers, and related fiber and articles of manufacture. 5
- It is known that extrusion processing of thermoplastic polymeric materials (the term referring, for example, to amidic or esteric or hydrocarbon structure polymers) involves the use of dies including an endless screw, or auger, arranged to rotate within a heated drum for melting the thermoplastic material and simultaneously pumping it through the dies or into the molds. 10
- The thermoplastic polymeric material is usually fed in as chips or flakes, and is input with energy both in the form of melting thermal energy and of pumping mechanical energy.
- The molten polymer has sometimes an abnormal behaviour during the extrusion process, which may involve, for example, non-constant flow rates while leaving unchanged the die rpm, or varying rates totally unrelated to the die rpm. 15
- The reasons why the molten polymer behaves abnormally in the die are manifold: enhanced intermolecular cohesive forces which result in irregular mass ratios for a given flow rate, molten polymer-metal (forming the apparatus as a whole) adhesive forces which may result in an irregular behavior of flow rate as the screw speed changes. 20
- While the use of additives to assist a smooth outflow of a molten thermoplastic polymer through the extruder and improve the product characteristics has been long known, their application to textile fibers involves considerable difficulties, both on account of the moderate amounts which have to be introduced not to adversely affect the yarn properties and of the problem of homogenizing the additive with the thermoplastic material chips. 25
- It is an object of this invention to provide an additive which can impart improved extrudability characteristics to a thermoplastic material for textile fibers.
- A further object of this invention is to arrange for the additive provided to assist in improving the mechanical characteristics of the extruded material, while improving its stretchability. 30
- In fact, a noteworthy feature of a fiber obtained by means of a melt spinning process is stretchability, which controls the amount of material that can be processed on one die.
- In fact, during subsequent stretching processes it becomes possible, for a given final count, to process a process having an initial count, as achieved in spinning, which is the higher the higher is the product stretchability.
- Thus, spinning machines can, for a given picking speed, produce higher counts, and hence a larger product output per unit time. 35
- The behavior of the thermoplastic polymer during the extrusion process is studied, in this specific case, on small size extruders of the type of those employed with rheometry apparatus, capable of monitoring the polymer temperature at various points on the extruder screw, as well as the amount of power absorbed by the electric motor driving the screw and the material flow rate as the rpm change. The ideal behavior of a polymer during extrusion processes requires but low energy inputs and flow rates which grow linearly with the extruder screw determining the overall rate of flow. 40
- It is generally observed that where the polymeric material has an irregular behavior at the rheometer die, that affected by larger size dies, such as used with melt spinning, is likewise irregular. 45
- A smooth rate of flow ensures proper treatment by extrusion and avoids that a part of the material may stay for too long in the die, with the attendant risk of local overheating and thermal degradation of the polymer macromolecular chains and consequent loss of product quality. Furthermore, a smooth flow of the melt through the die allows for the electric motor to operate effortlessly, thus reducing the electric power consumption. 50
- It is a primary object of this invention to provide a method which can overcome such prior drawbacks by providing a synthetic fiber spinning method by the extrusion of thermoplastic polymers, wherein said polymers are admixed prior to extrusion with a stearate-base additive.
- It has been unexpectedly found that a particular class of additives can make the behavior of molten materials through the dies a regular one, with considerable benefits also for an application to spinning systems where dies are utilized for mass transportation. 55
- Further, a polymeric material containing the cited additives exhibits particular structural characteristics in terms of stretchability of the thread-like product, enabling, other factors being the same, an increased output of the extruding equipment. 60
- The class of compounds is that of stearates, having an esteric or amidic chemical structure; preferred are the stearates which contain one or more stearyl and/or stearyl groups:  $X-NH-Y$ ,  $X-O-Y$ ,  $X-O-R-OX$ ,  $X-NH-R-NHX$ ,  $Y-NH-R-NH-Y$ ,  $Y-O-R-O-Y$ , where R is the steroyl group  $CH_3(CH_2)_{16}-CO$ ; Y is the stearyl group  $CH_3(CH_2)_{17}-$ ; R = alkyl, aryl, alkylaryl group.
- In particular, this invention contemplates the use of stearyl stearate as the additive, as is 65

brought out by the Examples hereinafter which have been conducted on experimental equipment, as indicated.

The amounts to be used vary from 0.005% to 1%, preferably from 0.03% to 0.3%, depending on the polymer being considered.

- 5 Textile fibers are highly responsive to the presence of additives, which may jeopardize their characteristics like dye-assuming ability, or color stability, etc. For this reason, it is extremely important that the additives be introduced in small percent amounts. The main problem is, therefore, that of ensuring thorough homogenization of a small amount of additive through the mass of polymeric material, to provide uniform characteristics for the products and avoid highly undesirable local build-ups. 5 10

The subject matter of this invention is the addition of an amount of an additive belonging to the cited classes and in the amounts indicated, to the mass of thermoplastic polymer prior to spinning, and in particular either before or during the drying step.

- Contemplated is a drying step for a thermoplastic polymer useful in the production of synthetic fibers which is conducted at temperatures in the range of about 110° to 130°C, in rotary driers which mix large amounts of polymeric material granules over a time of some hours. The addition of an additive from the cited class at this stage brings about undoubted advantages, the melting temperature of the additives in question being lower than that at which drying is conducted. Throughout the drying step, therefore, the additive is present in the fluid state, and can flow over the polymer granules to distribute itself over the latter in a uniform fashion in the form of a film. The good distribution of the additive is aided by the mixing to which the granules are subjected, which exerts a destructive action on any additive agglomerates. 15 20

- On exiting the drier, the granules of polymeric material have on their surfaces a very thin uniform film. 25

During the extrusion steps, the additive melts around the auger together with the polymer granules, and appears to be homogeneously distributed throughout the melt, thus facilitating the material extrusion.

- Other advantages connected with the use of the above-described substances, additionally to the regular operation of the die, concern the possibility of lowering the temperatures of the heating regions of said dies to provide a molten polymer of appropriate viscosity for the subsequent spinning operation. 30

The possibility of working at a lower temperature is advantageous with polymers which tend to degrade at the process temperatures.

- A further advantage, resulting from the presence of stearate-based additives, concerns the improved mechanical properties of the extruded material. 35

The finding forming the subject matter of this invention is described in the following Examples of some preferred embodiments thereof.

- 40 **EXAMPLE 1** 40

5 kg polyethyleneterephthalate containing 0.04% stearyl-stearate were vacuum dried down to a residual water value of 0.006%. The polymer is added under an anhydrous nitrogen stream into the hopper of a Brabender rheometer using a single-screw extruder having a diameter of 20 mm.

- Graph 1, where shown respectively on the ordinates are the polymer flow rates and on the abscissae the corresponding die rpm, shows a rectilinear behavior Q/n.g. Compare in that same graph, by contrast, the behavior of polyethyleneterephthalate not containing stearate; there exists a gap of irregular behavior in the 30 to 60 rpm range. 45

- The Example demonstrates, therefore, the importance of the additive in making the relation Q/n.g. linear. 50

Graph 2 shows instead the energy input, in Newtons per meter (NM), to the Brabender screw according to rpm.

- The presence of the stearate additive makes the behavior linear which would otherwise be irregular with much higher energy values. 55

#### EXAMPLE 2

5 kg Nylon 6 containing 0.1% N,N'-ethylenebisdistearamide are dried under a vacuum down to a residual water value of 0.08%. The polymer is fed in under an anhydrous nitrogen stream into the hopper of the Brabender rheometer.

- Graphs 3 and 4 show, respectively, the behaviors of Q/n.g and N.m/n.g in the presence and absence of the additive. 60

Also in that Example it is confirmed that the molten polyamide 6 undergoes a regular mass transport through the die, by varying at will the rpm, when the stearate additive is added in the amounts considered.

- Two samples of the polymer extruded at the rate of 70 rpm, respectively containing and not 65

containing distereamidic additive, show a crystallinity of 41% and 36% as determined on a Mettler instrument for differential thermal analysis, indicating improved crystallinity accompanied by improved mechanical properties of the extrudate.

A further series of analyses and tests have been conducted to demonstrate how the use of sterylstearate as an additive for textile fibers, results in an unexpected improvement of the product stretchability.

The tests are comparatively conducted on a common yarn sample (ST) and a sample containing sterylstearate in the cited percentages.

The cited improved characteristics of the polymer treated with the additive of this invention are described in the following series of analyses and tests, conducted comparatively on a yarn sample containing the additive and sample yarn of ordinary composition (ST).

It may be noticed that owing to the addition of the additive, improved stretchability can be achieved without prejudice for any of the yarn characteristics. The production output pattern, expressed as percent increase of the processed material through the same extruder per unit time, reaches 6-7%, thanks indeed to the cited stretchability increase.

### EXAMPLE 3

The tests are conducted at two different picking speeds: 4,000 and 4,500 m/minute. Data relating to the latter speed are shown in brackets in Table 1.

In Table 1, the additive employed is commercially available stearyl stearate.

		REFERENCE	ADDITIVE	
25	Count/filaments (dtex/number)	27.2/7 (27/7)	29.3/7 (28.7/7)	25
	Tenacity (cN/Tex)	45.6 (42.7)	42.7 (41.8)	
	Elongation (%)	72 (78)	77 (79)	
30	Initial module (cN/Tex)	136 (120)	117 (116)	30
	Count filaments (dtex/N)	21.9/7 (21.8/7)	22.2/7 (22/7)	35
	Tenacity (cN/Tex)	42.2 (43.5)	43.6 (43.8)	
	Elongation (%)	28.3 (28.9)	27 (28)	
40	Initial module (cN/Tex)	189 (190)	202 (198)	40
	Crimp rigidity (%) (1)	50 (55.5)	51 (51.2)	
45	Crimp shrinkage (%) (2)	62.5 (64.5)	62 (64)	45
	Crimp module (%) (2)	45.4 (45.9)	43 (45.6)	
50	Crimp stability (%) (2)	79 (74.8)	74 (73.8)	50
	K stretch	1.24 (1.24)	1.32 (1.30)	
55				55

TABLE I: comparison between the characteristics of Nylon 6 fiber containing and not containing 0.3 percent stearylstearate. The data in brackets relates to threads extruded at a speed of 4,500 m/sec.

(1) with the Heberlein method

(2) with the Texturmat method

1. A method of spinning synthetic fibers by the extrusion of thermoplastic polymers, characterized in that said polymers are admixed prior to their extrusion with a stearate-based additive.
2. A method according to Claim 1, characterized in that said additive is selected from
5. stearates having an esteric or amidic cyclic structure. 5
3. A method according to Claim 2, characterized in that said additive contains at least one or more stearyl or steryl groups.
4. A method according to Claim 3, characterized in that said stearyl and/or steryl groups are present in compounds having a general formula selected from  $X-NH-Y$ ,  $X-O-Y$ ,
- 10  $X-O-R-OX$ ,  $X-NH-R-NHX$ ,  $Y-NH-R-NH-Y$ ,  $Y-O-R-O-Y$ , where R is an alkyl, aryl, or alkylaryl group, and X is the stearyl group  $CH_3(CH_2)_{16}-CO-$ , and Y is the steryl group  $CH_3(CH_2)_{17}-$ . 10
5. A method according to Claim 1, comprising a step of drying said thermoplastic polymer upstream of said extrusion, wherein said admixture is performed concurrently with said drying
- 15 step. 15
6. A method according to Claim 5, characterized in that said additive is added in concentrations varying from 0.1% to 0.3%.
7. A method according to Claim 6, characterized in that said additive is stearylstearate.
8. A method according to Claim 6, characterized in that said additive is ethylenebistrisstearyl-
- 20 amide. 20
9. A method according to Claim 1, characterized in that said thermoplastic polymer is polyethyleneterephthalate.
10. A method according to Claim 1, characterized in that said thermoplastic polymer is polyamide 6.
- 25 11. A fiber obtained with the method according to any of the preceding claims, characterized in that it contains said additive. 25
12. A fiber according to Claim 11, characterized in that said additive is provided in concentrations varying from 0.03% to 0.3% by weight.
13. A fiber according to Claim 11, characterized in that the concentration of said additive
- 30 ranges from 0.1% to 0.3% by weight. 30